EFFECT OF La_2O_3 ADDITION ON SINTERING OF ALUMINA

A Thesis Submitted
in Partial Fulfillment of the Requirement
For the degree of
BACHELOR OF TECHNOLOGY

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CERTIFICATE

This is certified that the work contained in the project entitled "EFFECT OF La_2O_3 ADDITION ON SINTERING OF ALUMINA" by Mr. Pankaj Singh (Roll 10608035), have been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

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ACKNOWLEDGEMENT

I express my deep gratitude to my guide, Prof R.Sarkar for his valuable advice, time and guidance in the completion of this project work. My heartfelt thanks to all the faculty members for their suggestions during this project work. My sincere acknowledgement to the Research Scholars, M. Tech students and the non-teaching staff for the help and cooperation extended to us. And finally, my hearty thanks to all my friends who have constantly helped me.

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ABSTRACT

Densification of alumina has been studied in the present work in presence of La_2O_3. La_2O_3 as dopant has been added to the extent of 0.5, 1 and 2 weight % to pure alumina and studied for pressure-less sintering. Pellets of without and with La_2O_3 containing compositions were sintered at 1550^0C 1600^0C, 1650^0C and then characterized for densification, phase analysis and SEM & EDAX studies. In phase analysis $LaAlO_3$ has been observed in the sintered samples indicating reactions between alumina and dopant phase. Results reveal that La_2O_3 is not a beneficial sintering additive for alumina in the temperature range of 1550^oC to 1650^0C.

LIST OF FIGURES

LIST OF TABLE

CONTENTS

CHAPTER 1 ~ INTRODUCTION

1.1 Importance of alumina:

Alumina (Al_2O_3) is one of the most widely specified general purpose technical ceramics, it has wear resistant with high compressive strength even against extreme temperature and corrosive environments alumina is also excellent insulator and are gas tight, It is the most effective and widely used material in the family of engineering ceramics, It is also a hard ceramic material , hence used as the cutting tools.

Aluminium oxide is an amphoteric oxide of aluminium with the chemical formula Al_2O_3. It is also commonly referred to as **alumina** or **aloxite** in the mining, ceramic and materials science communities. It is produced by the Bayer process from bauxite. Its most significant use is in the production of aluminium metal, although it is also used as an abrasive due to its hardness and as a refractory material due to its high melting point. [4]

1.2 Properties of Alumina:

Key properties of alumina are

(a) High wear resistant

(b) Excellent dielectric properties

(c) Resistant to strong alkali and acid attack at elevated temperature

(d) Good thermal conductivity

(e) Excellent size and shape capability

(f) High strength and stiffness

(g) Available in purity range from 90% Al_20_3 to 99.5% Al_2O_3 for the most demanding high temperature application.

1.3 Alumina grades:

90-97% Alumina -Best suited for metallurgy because of the large grain structure.

98-99.5% Alumina-Commonly range of isostatically pressed grades with extruded shape also available at low cost.

Tolerance- As fired alumna's tolerance is generally only possible within a few percentage of dimension externally high tolerance are attainable but only by precise machining, the fired part using the diamond grinding technique.

1.4 Application of refractories:

(a) Refractory application

(b) Sodium vapour lamps

(c) Grinding media

(d) Abrasion resistant tube

(e) Thermometry sensors, laboratory instrument tube and sample holders, instrumentation parts for thermal property test machines.

(f) Ballistic armor

(g) Electronic substrate

(h) Thread and wire guide

(i) Furnace lanner tube high temperature electrical insulators

(j) Seal rings

(k) Wear pads

(l) Gas laser tubes

(m) High voltage insulators

1.5. Extraction Routes of Alumina through Bayer's process:

'Bayer' process is the most common process for the extraction and purification of alumina , In the first step of the process ground bauxite is mixed into a solution of sodium hydroxide. Now applying steam and pressure in tanks containing the mixture, the bauxite slowly dissolves. The alumina released reacts with the sodium hydroxide to form sodium aluminate. After the contents of the tank have passed through other vessels where the pressure and temperature are reduced and impurities are removed, the solution of sodium aluminate is placed in a special tank where the alumina is precipitated out. The precipitate is removed from the tank, washed, and heated in a kiln to drive off any water present. The residue is a commercially pure alumina. [8]

$Al_2O_3 + 2\ OH^- + 3\ H_2O \rightarrow 2\ [Al(OH)_4]^-$

$2\ Al(OH)_3 \rightarrow Al_2O_3 + 3\ H_2O$

1.6. Sintering of alumina:

Sintering of alumina is done in two ways .

(a) Solid state sintering where we need purity more than 99.7%.

(b) Liquid state sintering where we need purity from 80-99.7%.

Solid state sintering requires exclusively for translucent Al_2O_3 ceramics, because presence of pores arises scattering of visible light which from opaque alumina to get to get a very pure alumina body, sintering can be done by 3 solid state process, we require high temperature (>1900°C) ,still the body contains 5% porosity and abnormal grain growth.

To overcome this problem Alumina is sintered using 0.25 Wt% of MgO, and sintering at the temperature range of 1700-1800°C in H_2 atmosphere, to make the body translucent.

BY the microstructural observations we revealed that MgO eliminates the discontinuous grain growth of Al_2O_3, and grain boundaries do not break away from pores ,Which prevents the inclusion of pores trapped inside the new grain with long diffusion path for densification.

The majority of MgO doped into alumina resides at the grain boundaries because the dissolution of MgO in alumina is small, This leads to solute drag mechanism which reduces the boundary mobility and hence prevents abnormal grain growth, and pores also are attached to the boundary that's why pores can be easily mobile.

1.7. Effect of La_2O_3 on Alumina:

La_2O_3 is added in alumina as sintering aid, which may bring down temperature of sintering by creating stearic hindrance in alumina structure, solid solution forms when they follow the Hume-Rothery rule.

Hume Rothery rule: (Substitutional solid solution):

(a) Valency should be same

(b) Electro negativity should be similar

(c) Size difference should be within 15%.

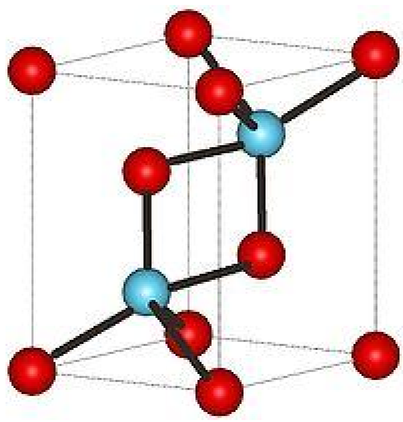
But here although valency state of Al^{3+} and La^{3+} is same, but there is so far difference between the electro negativity and atomic radii of La^{3+} and Al^{3+} .We have atomic radii of La^{3+} and Al^{3+} are o.103 and 0.053 nm respectively, La_2O_3 preferably exists at alumina grain boundaries, where the concentration distribution of La_2O_3 is thermodynamically non-equilibrium, The movement of grain boundaries is blocked by La_2O_3 additive, so the pores can not be trapped into alumina grains and in such a way all the pores eliminated.[3]

1.8. To study the effect of La_2O_3 at the three different percentage of additive at three different temperature------

We have three batches which contains different wt% of La_2O_3 as additive in 50 gms of alumina and Al_2O_3 batches, containing 0.5 wt%, 1 wt% and 2wt%, one batch of 50 gms of 100% pure alumina or 0% La_2O_3 is made which helps to find the difference between the above batches after sintering at different temperatures, 1550,1600 and 1650°C, The soaking time at pick temperature in each process is 2 hours.

1.10. Lanthanum oxide:

Lanthanum oxide is metallic oxide of rare earth element lanthanum, Chemically it is found in La^{3+} state in nature and It is used to develop ferroelectric materials, and in optical materials.



1.11. Structure of lanthanum oxide:

La_2O_3 has an α-M_2O_3 hexagonal crystal structure at room temperature. In this hexagonal crustal structure La^{3+} metal ions are surrounded by a 7 coordinate group of O^{2-} ions, metal ions remains in the octahedral voids of oxygen ions which occupies the corners of octahedron . Now at high temperatures α-La_2O_3 converts to a γ-M_2O_3 cubic crystal structure, and it becomes cubic crystal structure, and this type of cubic crystal structure the La^{3+} ion is surrounded by a 6 coordinate group of O^{2-} ions. [2]

1.12. Effect of La_2O_3 on the Al_2O_3 sintering:

In the experiment different amount of La_2O_3 is used as sintering aid as 0.5%, 1% and 2 wt% to find the sintering effect with respect to 0% batches of pure reactive alumina at different temperature 1550,1600 and 1650°C .It is found that La_2O_3 from can not make any solid solution (subsitutional or interstitial Solid) of Al_2O_3 and La_2O_3,which can be described by Hume Rothery rule. Since to make any solid solution the valency state should be same, electro negativity should be same ,and there should be not more than 15% in the difference of radii of ions, but here electro negativity of La and Al is different ,and also there is a large difference between the radii of La and Al. and hence they can’t make solid solution.

Mechanism:

As ionic radii of La^{3+}(o.103 nm) is much larger than the ionic radii of Al^{3+} ions .hence when we add La_2O_3 in $Al_2O_{3,}La^{3+}$ enters in Al_2O_3 structure by replacing Al^{3+} ions. So it creates stearic hindrance which causes the increase in strain energy and due to increase in strain energy Al_20_3 sinters even at lower temperature (means about 1550°C).

where the concentration distribution of La_2O_3 is thermodynamically non–equilibrium, The movement of grain boundary is blocked by La_2O_3 additive ,so that pores could not trapped into alumina grains, when the migration speed of grain boundaries V_b is equal to V_p ,the migration speed of pore, pore would stay at the grain boundaries all along and move together with grain boundaries, Pores would get together rapidly and eliminate from grain boundaries, which are the fast transfer channels of vacancy, As a result alumina grains, with almost no pores trapped in and become fine and pores.[7]

CHAPTER 2~ LITERATURE REVIEW

2.1 What is sintering?

Thermal treatment that bonds particles together into a solid, coherent structure by means of mass transport mechanisms occurring largely at the atomic level.

Mass movements which occur during sintering consist of the reduction of total porosity by diffusion followed by material transport, mostly density of a collection of grains increases as material flows into voids, causing a decrease in overall size.

The initial powder (green body) has a large surface area relative to it's volume, this surface area relative to it's volume, this surface area provides the driving force in sintering, which is the reduction of free surface energy resulting from the surface area of the particles.

2.2 Different types of sintering:

Sintering basically is of two types…..

(a) Solid state sintering

(b) Liquid assisted sintering

2.3 There are different stages of sintering in both types of sintering are described below:

1. Initial stage of sintering:

(a) Initially there is formation of local point of fusion without shrinkage of compact.

(b) After some time as temp increases there is a neck formation at the contact point, with the resulting concave curvature at the neck.

2. Intermediate stage of sintering:

(a) There is formation of necks, and neck growth

(b) Pores forming arrays of interconnected cylindrical channels.

(c) Particles centers approaching are another, with the resulting compact shrinkage.

(3) Final stage of sintering

(a) Isolation of pores i.e. relative densityexceeding-93%..

(b) Elimination of porosity

(c) Grain growth

2.4. Mechanism of solid state sintering:

Polycrystalline material sinter by diffusional transport of matters whereas amorphous materials sinter by viscous flow, in polycrystalline materials matter transport takes place along definite paths that define the mechanism of sintering, matter transport takes place along definite paths that define the mechanism of sintering ,matter is transported from the regions of higher chemical potential to region of lower chemical potential ,there are six different mechanism of sintering in polycrystalline materials.

(a) Surface diffusion

(b) Lattice diffusion from surface

(c) Vapour transport

(d) Grain boundary diffusion

(e) Lattice diffusion from grain boundary

(f) Plastic flow

Surface diffusion , lattice diffusion and vapour diffusion are the only mechanism which leads to the actual densification but all causes the neck to grow and so influences the rate of densification. While the amorphous materials viscous flow leads to neck growth as well as the densification.

2.5. Explanation of solid state sintering:

To enhance the slow in diffusion the one are enhanced or encouraged through the following method.

(a) Chemical doping

(b) Atmospheric control

(c) An appropriate time per temperature cycle

2.6. Sintering of Al_2O_3 with aid of MgO:

MgO is added into the Al_2O_3 with a very small amount 0.25 wt% of Al_2O_3 content, this allow the achievement of fine, grained material at full density microstructure studies revealed that MgO eliminates the discontinuous grain growth of Al_2O_3 grains, The grain boundary do not break away from the pores ,which prevents the inclusion of pores trapped inside new large grains, with slow/long diffusion path densification.

The mechanism by which MgO slowdown grain boundary movements in alumina could be the follows.

(a) The majority of MgO doped into Al_2O_3 resides at the grain boundaries, because the dissolution of MgO in Al_2O_3 is small or 300 ppm, This is due to the relatively large difference in ionic radius , 0.72 À for Mg^{2+} and 0.53 À for Al^{3+} .

(b) Any fast migration of the grain boundary would have to incorporate Mg^{2+} ions into the Al_2O_3 lattice, will the resulting increase in internal energy, unless a new compound spinel forms.

CHAPTER 3~ EXPERIMENTAL

3.1. Pellet preparation

First alumna powder was taken and mixed with different amounts of La_2O_3 as per compositions. Next these powders were mixed in dry condition and then 2% PVA solution was added to it as green binder. After mixing it for 5 min, the material is sieved 3 times by 500μm sized sieve so that we can get proper mixing. Next each compositions were pressed to pellet (each 2.2 gm) using a high chromium steel mold and punch. Hydraulic Press (Carver, USA, make) was used for this purpose and a pressure of 1.5 ton was used on 1.2 mm^2 area with a holding time of 60 sec. Stearic acid is used as lubricant.

3.2. Bulk Density and apparent porosity Measurement

The bulk density and apparent porosity of the sintered pellets were determined by Archimedes principle using water. Dry Weight is measured and then the pellets were kept in distil water and then vacuuming is done in place of boiling for about 45 min-1 hr. After that suspended weight is measured using apparatus in which pellet is suspended in water and weight is measured. After taking suspended weight, soaked weight is taken. Hence the dry weight, soaked weight and suspended weight were measured. The bulk density and apparent porosity were calculated by the formulas:

Bulk Density = dry weight **/** (soaked weight – suspended weight)

Apparent porosity= (soaked weight-dry weight) **/** (soaked weight – suspended weight) x 100

3.3 Volume Shrinkage

Volume shrinkage can be simply calculated by the formula….

Volume shrinkage = $(V_B - V_A)/ V_B \times 100$

Here, V_B = Volume of the pellet before heat treatment

And V_A =Volume of the pellet after heat treatment

3.4. Materials used:

(a) Alumina (Commercial grade, Reactive alumina A17 NE)

(b) La_2O_3 powder (AR grade)

(c) 2% PVA solution

(d) Stearic acid

(e) Acetone

3.5. Apparatus required:

(a) Weighing machine

(b) Petri dishes for mixing sample

(c) Sieve of 500 mesh size

(d) Measuring cylinder

(e) Die and hydraulic Press

3.6. Experimental procedure:

Step 1:

Four different batches of each 50 gms of Al_2O_3 (Reactive alumina made by ALMATIS GERMAN) were taken.

Step 2:

Now these 4 batches 0%, 0.5%,1%, and 2% of La_2O_3 is added as o gmms,0.25 gms,0.5 gms and 1 gms respectively in each batch, such that whole batches forms a combine weight of 50 gms.

Step 3:

PVA solution (Polyvinyl alcohol) of 2% is prepared

Step 4:

Then in each batch 3 ml or 2% PVA solution is added , after mixing it for 5 minutes the material is sieved 3 times by 500 μm sized sieve, so that we can get proper mixing.

Step 5:

Now 2-2 gms of sub batches are formed to make pellets with a die pressed by hydraulic press, at pressure of 1.5 tonne on 1.2 mm area at a holding time of 60 sec, using the stearic acid or lubricant.

Step 6:

After preparing the pellets from each package/batches ,these pellets are dried for 24 hours at 110°C.

Step7:

After drying , the dimensions (Height and diameter) of each pellet has been taken with the help of vernier caliper.

Step 8:

Now from each batch 3 pellets were taken , now it is sintered at 1550°C temperature, with soaking time of 2 Hours.

Step 9:

Now same process (step 8) is done for other 24 pellets at temperature of 1600 and 1650°C.

Step 10:

The sintered pellets are taken out, and bulk density, apparent porosity and shrinkage is calculated.

Step 11:

Now XRD and SEM is also done to study the phase present and microstructure in the pellets.

CHAPTER 4 ~ RESULTS AND DISCUSSIONS

4.1 Volume shrinkage of Sintered Samples

Table no. 1(Calculation of volume shrinkage at 1550°C)

Material used (sample No.)		Green Dimension (Initial)			Dimension after heat Treatment (1500°C)			Volume Shrinkage (%)	Average Volume Shrinkage (%)
		Diameter (mm)	Height (mm)	Volume (mm)	Diameter (mm)	Height (mm)	Volume (mm)		
La_2O_3 (0%)	1	12.01	6.79	769.2	11.20	6.35	625.60	18.66	19.18
	2	12.01	6.84	774.87	11.16	6.37	623.10	19.58	
	3	12.01	6.71	760.15	11.16	6.27	613.32	19.31	
La_2O_3 (0.5%)	1	12.01	7.14	808.90	11.30	6.78	680	15.93	16.19
	2	12.01	7.09	803.2	11.29	6.74	674.74	15.99	
	3	12.01	7.11	805.46	11.27	6.73	671.30	16.65	
La_2O_3 (1%)	1	12.01	7.11	804.12	11.31	6.76	679.14	15.54	15.42
	2	12.01	7.10	803.00	11.33	6.75	680.53	15.25	
	3	12.01	7.02	793.95	11.31	6.68	671.11	15.47	
La_2O_3 (2%)	1	12.01	7.14	808.86	11.35	6.82	690.03	14.69	14.75
	2	12.01	7.08	802.00	11.35	6.72	679.90	15.22	
	3	12.01	7.14	808.86	11.34	6.84	690.83	14.34	

4.2. Volume shrinkage of Sintered Samples

TABLE NO.2 (CALCULATION OF VOLUME SHRINKAGE AT 1600°C)

Material used (sample No.)		Green Dimension (Initial)			Dimension after heat Treatment			Volume Shrinkage (%)	Average Volume Shrinkage (%)
		Diameter (mm)	Height (mm)	Volume (mm)	Diameter (mm)	Height (mm)	Volume (mm)		
La_2O_3 (0%)	**1**	12.01	6.75	764.68	10.86	6.21	575.22	24.78	24.48
	2	12.01	6.69	757.88	10.94	6.15	578.09	23.72	
	3	12.01	6.71	760.15	10.85	6.17	570.47	24.95	
La_2O_3 (0.5%)	**1**	12.01	7.15	810.00	11.10	6.61	639.64	21.03	21.47
	2	12.01	5.03	569.80	11.13	4.49	436.84	23.33	
	3	12.01	7.12	806.60	11.10	6.58	636.73	21.06	
La_2O_3 (1%)	**1**	12.01	7.10	804.32	11.09	6.56	633.66	21.21	21.16
	2	12.01	7.11	805.46	11.10	6.57	635.77	21.06	
	3	12.01	7.09	803.19	11.09	6.55	632.69	21.22	
La_2O_3 (2%)	**1**	12.01	7.10	804.32	11.16	6.56	641.68	20.22	20.56
	2	12.01	6.66	754.48	11.15	6.12	597.57	20.79	
	3	12.01	7.07	800.93	11.13	6.53	635.32	20.67	

4.3 Volume shrinkage of Sintered Samples

TABLE NO.3 (CALCULATION OF VOLUME SHRINKAGE AT 1650°C)

Material used (sample No.)		Green Dimension (Initial)			Dimension after heat Treatment			Volume Shrinkage (%)	Average Volume Shrinkage (%)
		Diameter (mm)	Height (mm)	Volume (mm)	Diameter (mm)	Height (mm)	Volume (mm)		
La_2O_3 (0%)	**1**	12.01	6.78	768.07	10.86	6.11	565.96	26.31	27.69
	2	12.01	6.81	771.47	10.68	6.12	548.27	28.93	
	3	12.01	6.78	768.07	10.73	6.13	554.30	27.83	
La_2O_3 (0.5%)	**1**	12.01	6.59	746.55	10.87	5.93	550.30	26.28	25.15
	2	12.01	6.90	781.67	10.90	6.35	592.53	24.19	
	3	12.01	7.13	807.72	10.92	6.47	605.95	14.98	
La_2O_3 (1%)	**1**	12.01	6.92	783.93	10.88	6.37	592.22	24.45	25.20
	2	12.01	7.03	796.39	10.91	6.30	588.95	26.04	
	3	12.01	7.13	807.72	10.90	6.48	604.66	25.13	
La_2O_3 (2%)	**1**	12.01	7.24	820.18	10.92	6.31	590.96	27.94	26.50
	2	12.01	7.11	805.48	10.98	6.32	598.42	25.70	
	3	12.01	7.04	797.53	10.94	6.29	591.25	25.86	

4.4 Estimation of Bulk density and apparent porosity of samples:

TABLE 4 (AT 1550°C):

Material used (Sample no.)		Dry weight		Suspended wt		Soaked weight		Apparent Porosity	Bulk Density
La_2O_3 (0%)	**1**	1.8503	1.8434	1.3762	1.3745	1.9951	1.9871	23.45	3.01
	2	1.8557		1.3730		2.0040			
	3	1.8243		1.3695		1.9871			
La_2O_3 (0.5%)	**1**	1.9216	1.9136	1.4256	1.3762	2.0909	2.0820	25.43	2.89
	2	1.9096		1.4223		2.0780			
	3	1.9095		1.4122		2.0771			
La_2O_3 (1%)	**1**	1.9195	1.9168	1.4288	1.4238	2.0922	2.0882	25.75	2.88
	2	1.9165		1.4186		2.0918			
	3	1.9145		1.4243		2.0808			
La_2O_3 (2%)	**1**	1.9236	1.9250	1.4275	1.4274	2.1064	2.1070	26.81	2.81
	2	1.9231		1.4275		2.1013			
	3	1.9282		1.4273		2.1144			

4.5 Estimation of Bulk density and apparent porosity of samples:

TABLE 5 (AT 1600°C):

Material used (Sample no.)		Dry weight		Suspended wt		Soaked weight		Apparent Porosity	Bulk Density
La_2O_3 (0%)	1	1.8550	1.8493	1.3762	1.3712	1.9573	1.3978	15.61	3.26
	2	1.8409		1.3638		1.9332			
	3	1.8520		1.3712		1.9230			
La_2O_3 (0.5%)	1	1.9133	1.7114	1.4164	1.2693	2.0413	1.8258	20.50	3.07
	2	1.3035		0.9698		1.3920			
	3	1.9134		1.4218		2.0423			
La_2O_3 (1%)	1	1.9067	1.8965	1.4020	1.4014	2.0393	2.0289	19.23	3.02
	2	1.8996		1.4101		2.0359			
	3	1.8832		1.3923		2.0115			
La_2O_3 (2%)	1	1.9111	1.8710	1.4184	1.3917	2.0465	2.0004	21.25	3.07
	2	1.8006		1.3423		2.9233			
	3	1.8710		1.4146		2.0316			

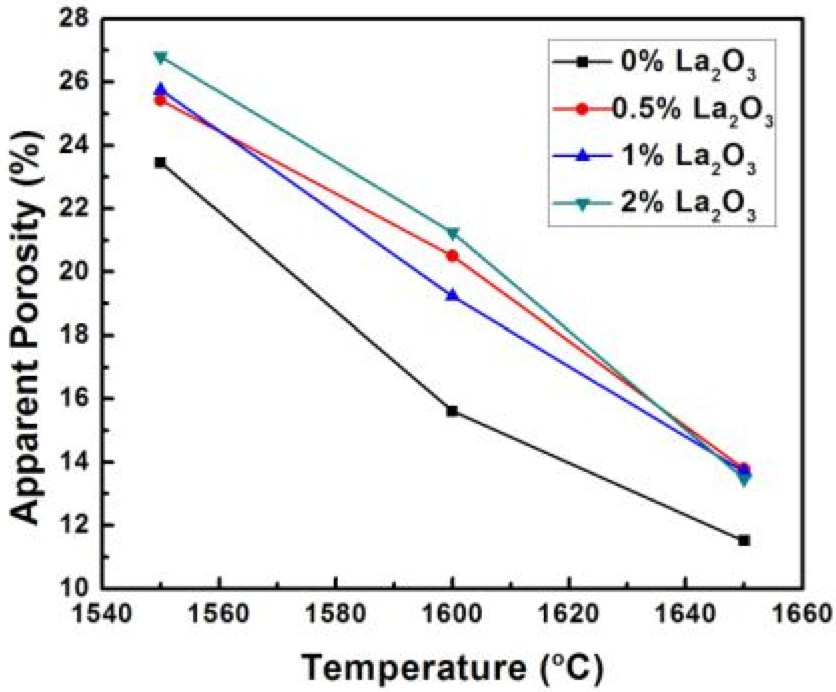
4.6. Estimation of Bulk density and apparent porosity of samples:

TABLE 6 (AT 1650°C):

Material used (Sample no.)		**Dry weight**		**Suspended wt**		**Soaked weight**		**Apparent Porosity**	**Bulk Density**
La_2O_3 (0%)	**1**	1.8491	1.8553	1.3612	1.3648	1.9202	1.9191	11.51	3.34
	2	1.8492		1.3586		1.9127			
	3	1.8677		1.3746		1.9244			
La_2O_3 (0.5%)	**1**	1.8266	1.8725	1.3380	1.3725	1.8872	1.9525	13.79	3.29
	2	1.8723		1.3746		1.9598			
	3	1.9187		1.4050		2.0107			
La_2O_3 (1%)	**1**	1.8815	1.8906	1.3757	1.3867	1.9480	1.9706	13.70	3.23
	2	1.9169		1.4039		1.9938			
	3	1.8736		1.3806		1.9700			
La_2O_3 (2%)	**1**	1.9193	1.9148	1.4098	1.4074	2.0080	1.9938	13.47	3.26
	2	1.8936		1.3885		1.9652			
	3	1.9311		1.4239		2.0082			

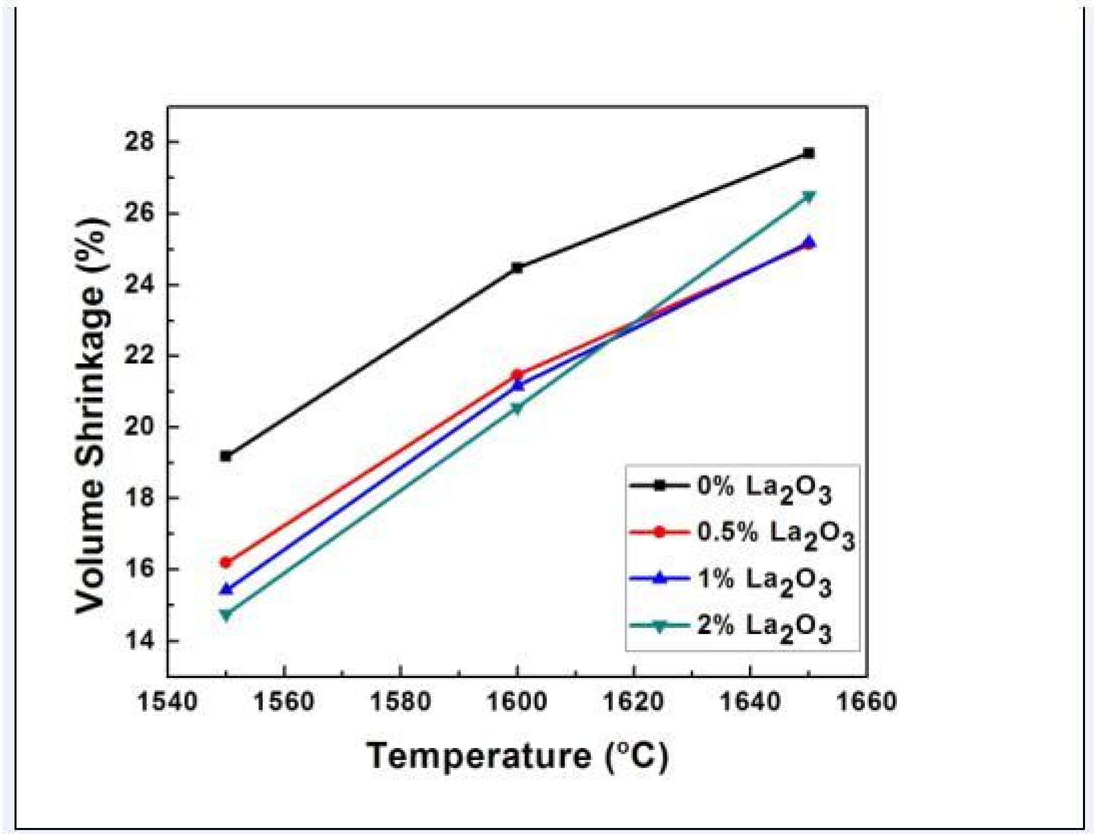
4.7. Volume Shrinkage, Apparent porosity and Bulk density :

(a) Apparent porosity Vs Temperature:



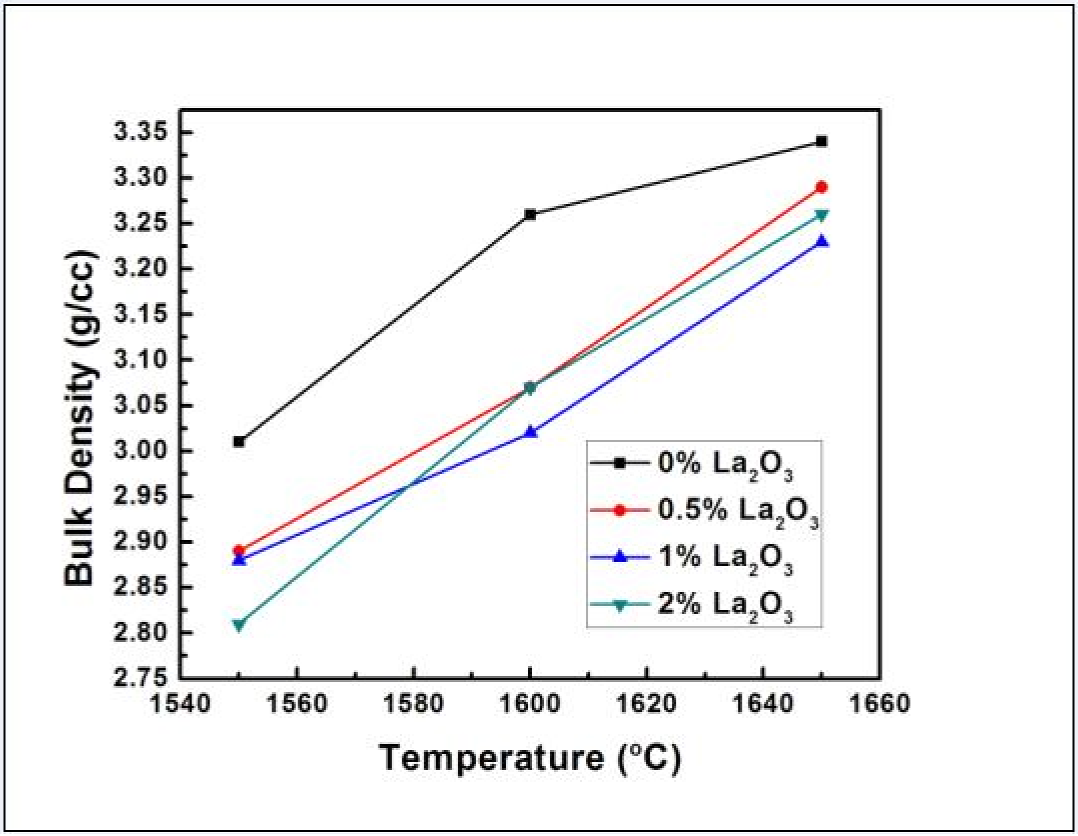
Apparent porosity decreases with increase in sintering aid and increase in temperature, this might be occur due to the formation of lanthanum aluminate phase.

(b) Volume Shrinkage Vs Temperature:



Volume shrinkage decreases with increasing the amount of sintering aid, and increasers with temperature.

(c) Bulk Density Vs Temperature:

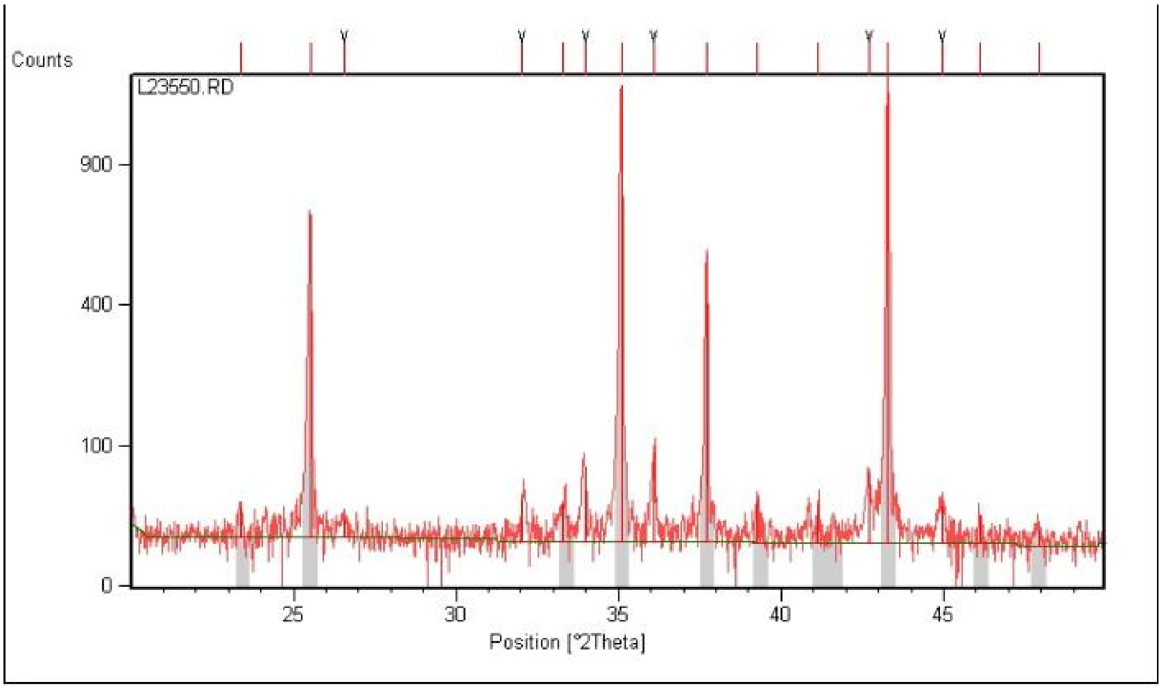


For each composition bulk density increases with increasing the temperature, but decreases with increasing the amount of sintering aid for every temperature. Hence it may be commented that it is not beneficial to use lanthanum oxide as sintering aid.

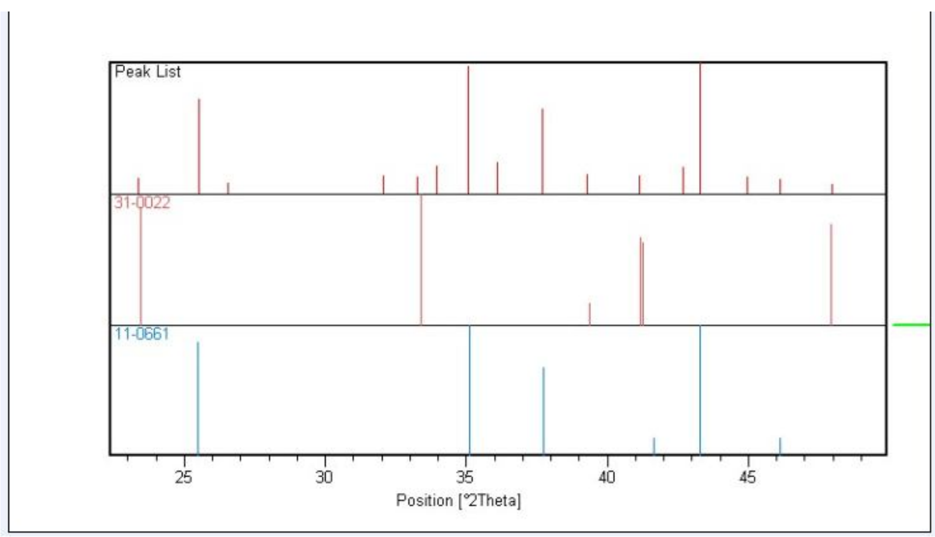
4.8. XRD Analysis:

(a) La_2O_3,2%,1550°C sample:

(i)



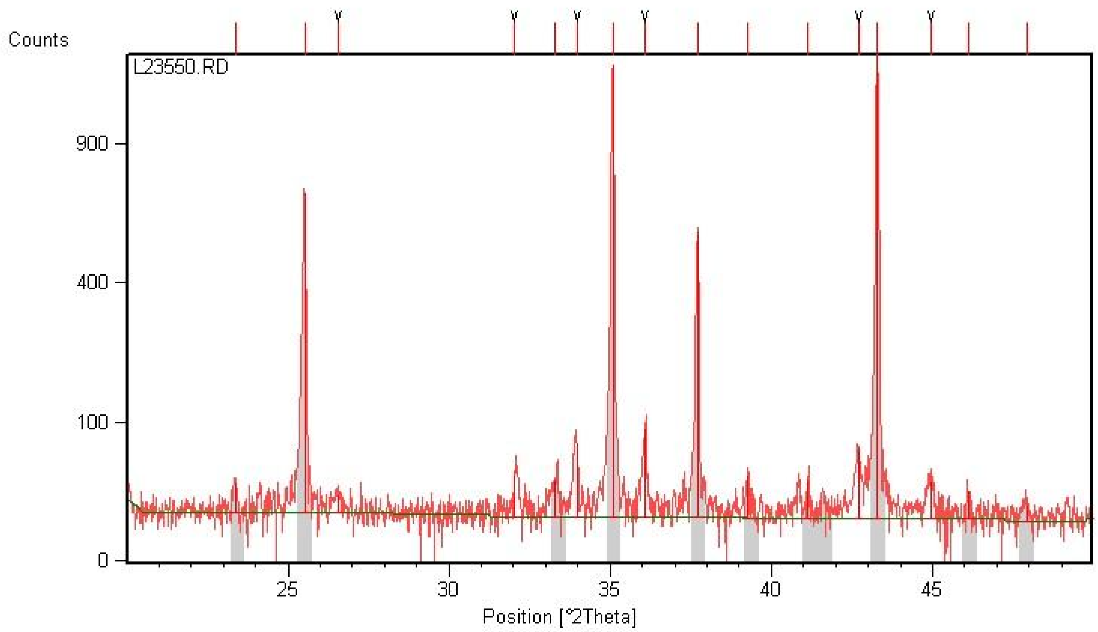
(ii)



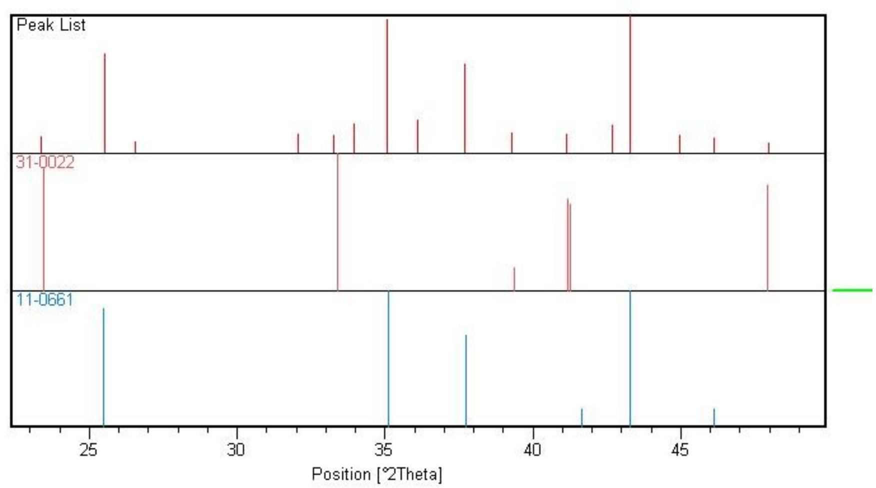
Here 31-0022 is lanthanum aluminate phase ($LaAlO_3$) and 11-0661 is aluminum oxide.

(b) XRD plot of 2% La_2O_3 doped alumina at 1650°C:

(i)

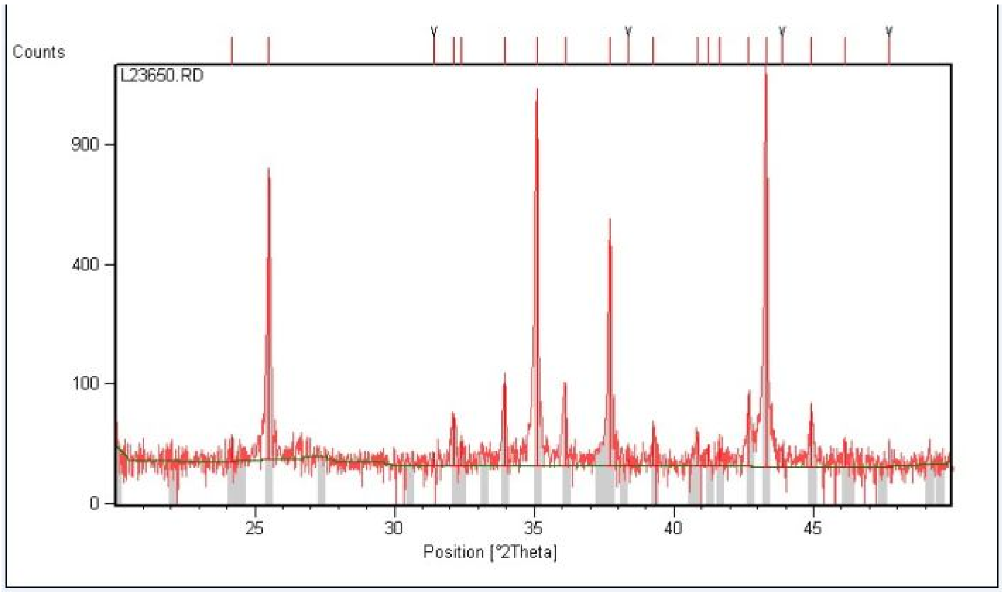


(ii)

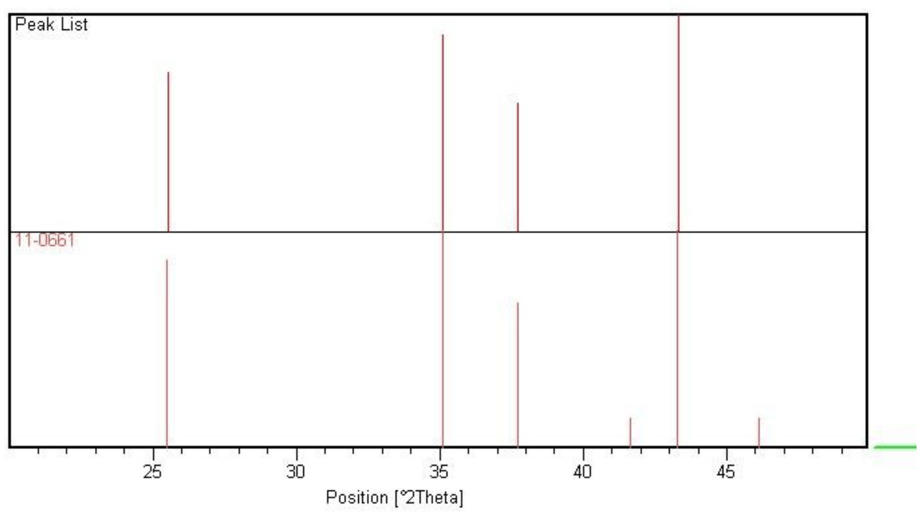


(c) XRD plot of 0% La_2O_3 doped alumina at 1650°C:

(i)



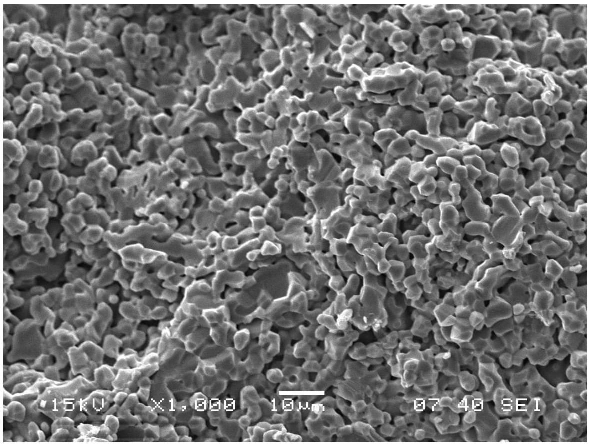
(ii)

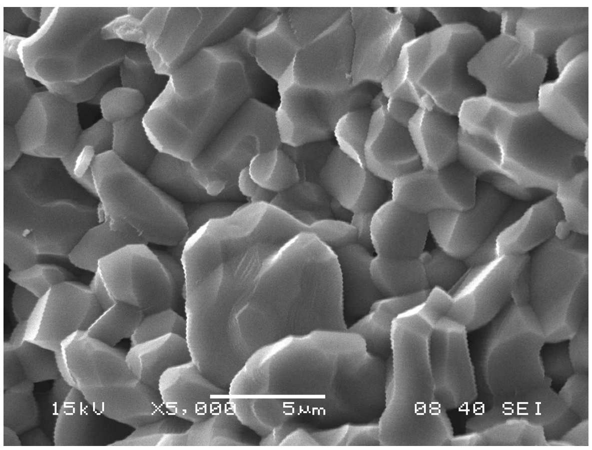


4.9

4.9. Scanning Electron Microscopy (SEM)

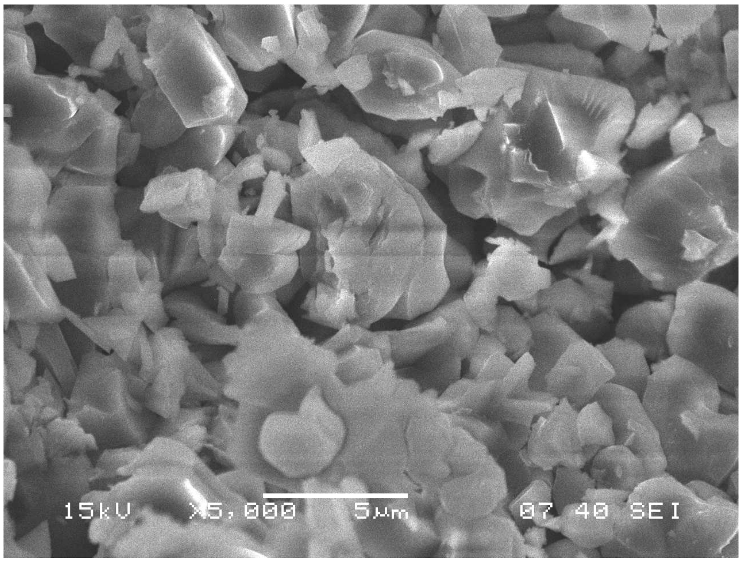
(a) SEM of 0% La_2O_3 doped Al_2O_3 sample at 1650°C

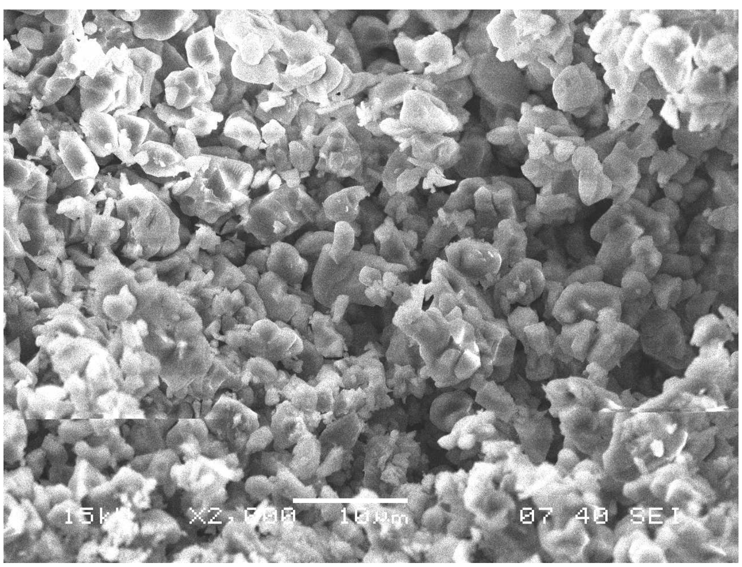




SEM photomicrograph shows that the grains of pure alumina sintered at 1650°C is not highly compacted and pores are commonly observed. Also there is a wide variation in the grain sizes.

(b) SEM image of 2% La_2O_3 doped Al_2O_3 at 1650°C…….





SEM photomicrograph of 2% La_20_3 sintered at 1650°C shows distribution of uniform grains in a less compact manner, pores are commonly observed, grains are sharp and angular in nature.

CHAPTER 5~ CONCLUSION

(a)Reaction between alumina and La_2O_3 is occurring at high temperature forming $LaAlO_3$ phase.

(b)Bulk density, apparent porosity and shrinkage values indicate that lanthanum oxide is not a effective as dopant for alumina in the temperature range of 1550oC to 1650^0C.

(c) A decrease in densification is observed as amount of lanthanum oxide (additive) increases, this shows that La_2O_3 is not good as sintering aid.

CHAPTER 6 ~ SCOPE FOR FURTHER WORK

(a) Future work on the densification with La_2O_3 can be done at further higher temperature.

(b) Sintering study can be done by taking the oxides of rare earth elements.

(c) Other physic-mechanical properties can also be studied to understand the effect of additive on these properties.

REFERENCES

1. J. S. Reed, *Principles of Ceramics Processing*, 2nd edn. Wiley, New York (1995).

2. From link of Wikipedia http://en.wikipedia.org/wiki/Lanthanum(III)_oxide

3. from the link of wikipedia http://en.wikipedia.org/wiki/Hume-Rothery_rules

4. From the link of wikipedia http://en.wikipedia.org/wiki/Aluminium_oxide

5. W.D.Kingery,H.K.Bowen and D.R.Uhlmann,Introduction yo ceramics,2nd Ed,John Wiley & Sons,Singapore,1991

6..M.N. Rahaman,Ceramic processing,CRC Press,Taylor & Francis Group,FL,2007.

7. Bernard BBguin, Edouard Garbowski and Michel Primet, Stabilization of alumina by addition of lanthanum, *Applied Catalysis, 75 (1991) 119-132.*

8. From the link of Wikipedia http://en.wikipedia.org/wiki/Bayer_process

9. From the link of Wikipedia http://en.wikipedia.org/wiki/Sintering